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ate received from CICO 7/31/95

ate submitted to ChemRisk/Shonka and DOE 8/7/95

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LIQUID SAMPLING OF C-616

Liquid Sampling of Large Drums

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Serial A-3601
File Index
Date August 15, 1946

CLINTON ENGINEERING WORKS

CARBIDE AND CARBON CHEMICALS CORPORATION

Laboratory Division

Works Laboratory Department

021

LIQUID SAMPLING OF C-616

Liquid Sampling of Large Drums

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Sampling of large drums of C-616

Outline

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- B. Statement of solution

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Summary of the Report

The validity of vapor phase techniques for the sampling of C-616 has long been questioned. It is clear that when a vapor transfer is made, a distillation of the material occurs. Since the impurities (primarily HF and fluorocarbons) in the C-616 do not have the same volatility as the C-616, the concentration of these substances in the distillate will differ from their concentration in the original sample. If liquid techniques were to be used it was felt that they would be more representative.

In an effort to investigate thoroughly the problem of liquid sampling it was first necessary to devise systems and techniques which would give good liquid samples. This report deals with this phase of the problem.

Liquid Sampling of Large Drums of C-616

I Summary

A. Statement of the problem.

This part of the general report on "Liquid Sampling of C-616" primarily concerns the development of systems and techniques suitable for use in sampling large drums of liquified C-616. The work was carried out using depleted feed material from the K-131 building of the K-27 area.

B. Statement of solution.

The system developed to "liquid sample" these drums was a small volume copper type, which could be attached to the valve of the drum, and which had one outlet to the sample receiver, one outlet to a vacuum pump and nitrogen line, and one arm with a bronze-tube Duragauge. The system was completely wrapped with a asbestos covered nichrome wire, and the temperature of the system controlled by a Variac.

II Definitions

A.

Drums referred to as C&CCC drums are small containers fitted with one 3/4" needle Kerotest valve in one end, and which hold approximately 350 pounds of C-616.

B.

Drums referred to as "A" type drums are large Chemical Warfare Service chlorine cylinders having two 3/4" needle Kerotest valves in one end, one valve being at the center and one valve being at the edge of the drum head. These containers hold approximately 4500 pounds of C-616.

C.

Harshaw cells are small nickel cylinders fitted with one 3/8" MP X 1/2 " SAE Kerotest diaphram right angle valve, and having a center well extending into the cell for about three-fourths of the cell's length. These cells hold approximately 5.5 pounds of C-616.

D.

The normal code symbols for materials are used throughout this report.*

* Enrichment Codes by W.C.Beard. 6/20/46.

III Solution of the problem.

A. Construction details

1. System for C&CCC drums.

The diagram for this system is shown in figure 1. The key to this diagram is given below:

a. Female nut for 3/4" Needle Kerotest valve. Uses a PTFE gasket.

b. Nickel 1/2 " Sweat cross at center of system. Nickel was used wherever possible to give strength to the system.

c. 1/2" SAE X 1/2" SAE Kerotest diaphram valve. The seat side is towards the cross.

d. 3/8" MP X 1/2" SAE right angle Kerotest diaphram valve. The seat side is towards the cross.

e. 6" bronze-tube Duragauge graduated from 30" mercury vacuum to 15 pounds per square inch pressure.

f. 1/2 " Sylphon flexible hose leading to vacuum pump and G-74 manifold.

g. Male fitting for 3/4" Needle Kerotest valve. Uses a PTFE gasket.

h. 1/2" Sylphone flexible hose 2-1/2 feet long leading to the Harshaw Cell.

i. Male portion of a 1/4" Nickel pipe union on the end of the sylphon hose.

j. Adapter for Harshaw cell, having the female portion of a 1/4" Nickel pipe union on one end and a 1/2" SAE flare nut on the other.

k. Harshaw cell.

l. 3/4" Needle Kerotest valve on the C&CCC drum.

m. C&CCC drum on wooden framework.

n. Wooden framework to hold C&CCC drum at approximately a 20° angle and about 36" from the floor.

o. Hot water bath for melting the material in the C&CCC drums prior to sampling.

The system from the C&CCC drum to the Harshaw cell, gauge, and vacuum line, is wrapped with asbestos covered nichrome wire, and this covered with asbestos ribbon. The Harshaw cell valve, well, and adapter are heated by means of two 250 watt in-fra-red heating bulbs.

2. System for A type drums.

The diagram for this system is shown in Figure 2. The key to this diagram is given below:

a. Female nut for 3/4" Needle Kerotest valve.

b. Nickel 1/2" Sweat cross at center of system, Nickel fittings were used where possible to strengthen the system.

c. 3/8" MP X 1/2" SAE right angle Kerotest diaphram valve. The seat side is towards the cross.

d. 6" Bronze-tube Duragauge graduated from 30" mercury vacuum to 15 pounds per square inch pressure.

e. 1/2" SAE X 1/2" SAE Kerotest diaphram valve. The seat side is towards the cross.

f. Male fitting for 3/4" Needle Kerotest valve soldered on the system.

g. Thickly insulated sylphon flexible hose leading to vacuum and G-74 manifold.

h. Sylphon flexible hose about 2 1/2' long leading to the Harshaw cell.

i. Male portion of a 1/4" Nickel pipe union on the end of the sylphon hose.

j. Harshaw cell adapter, with the female portion of a 1/4" nickel pipe union on one end and a 1/2 " SAE flare nut on the other.

k. Harshaw cell.

l. Center valve on the drum head. This is a 3/4" Needle Kerotest valve.

m. A 3/4" Needle Kerotest valve on the side of the drum head.

n. Drum head of type "A" drum.

o. Top section of hot circulating water heating jacket.

p. Bottom section of the hot circulating water heating jacket.

q. Crane hook for hoisting of the top portion of the heating jacket.

r. Valve manifold for vacuum and for G-74.

The system from the "A" drum to the Harshaw cell, gauge, and vacuum line is wrapped with asbestos covered nichrome wire and this covered with asbestos ribbon. The Harshaw cell, well, valve, and adapter are heated by means of two 250 watt infrared heating bulbs.

B. Conditions necessary for successful transfers.

1. Two conditions are necessary for liquid transfers:

a. The system is use must be completely leak tight. If this condition is not met, difficulty will be encountered.

(1) The operation will be hazardous to the workers, since C616 will readily escape into the room through even a minute leak.

(2) The obtaining of a liquid sample is made much more difficult, for air leaking into the evacuated system increases the internal pressure and may effectively block the flow of the liquid C-616.

(3) Because of the very ready hydrolysis of C-616, a leak introducing moisture may cause a serious plug of material or may freeze a valve.

(4) It is assumed that the large containers are leak tight when received.

b. The temperature must be controlled along the transfer system. A temperature gradient of 10 to 15 degrees Centigrade running from 75-80°C at the drum to a minimum of 65°C at the Harshaw cell.

(1) Since the melting point of C-616 is 64.05°C at 22.0 psia, any lower temperature along the system will cause a solid plug of C-616.

(2) Since the drum temperature is maintained at about 85-90°C, any higher temperature along the system might cause a sudden increase in pressure. This pressure increase may cause a vapor block which would prevent the passage of liquid C-616.

IV Field operating results.

A.

In sampling the C&CCC type drums the rate of taking samples started at four drums per eight hour shift. As proficiency was developed, this was gradually increased to eight per eight hour shift. Since April 29, 1946, when the maintainence and operations of the system was turned over to the Sampling Section, sixteen C&CCC drums per day on a two shift basis have been easily sampled.

B.

From the standpoint of the actual operation of the system for sampling "A" type drums, the same rate of sampling as for the C&CCC drums is possible, but since far fewer "A" drums are used and since only three preheating jackets are available, a maximum of approximately three drums in 48 hours can be sampled. While C&CCC drums require only three hours in the water bath to melt the contents, the A type drums require 36 hours or more to assure complete liquefaction

V Conclusions and further applications.

The problem of the liquid sampling of large volume drums of C-616 has been adequately solved. With the present rate of sampling by the Sampling Section, it is easy to supply the K-131 building with the amount of depleted feed material required.

This problem has definitely shown that the primary factor to be temperature control. Extensive work was done previously in trying to take liquid samples with a large 4.5 pound capacity pipette, heated in an oven. The failure of this venture is now attributed to inadequate heating and poor control of the temperaute gradient. It is believed that any liquid C-616 sampling problem can be handled by maintaining proper temperature control.

An appendix follows, giving the detailed operational procedure for the C&CCC drum sampling system. The same procedure, with slight modifications, is applicable to the system for sampling "A".

VI Appendix

A.

Detailed procedure for the operation of the system for liquid sampling of C&C CC type drums. The letters refer to the parts of the system as shown in figure 1 of this report.

Operation	Reason for Operation
1. While the C&CCC drum in the water bath is heating, the Harshaw cell (k) is attached by means of its adapter, and the system preheated. The variac must be set at about 75-80 volts and the infra-red lamps focused on the Harshaw adapter and well.	1. With temperature the primary considerations, adequate preheating is vital.

Operation	Reason for Operation
2. When the drum is above 75°C and the system above 65°C, the drum is lifted from its water bath and placed on its rack in such a position that the valve opening is down. The rack holds the drums at about 20° angle, insuring liquid being directly behind the valve as well as an appreciable liquid head.	2. The valve opening must be down so that the system will be properly aligned when attached to the drum.
3. The system is attached to valve "1" by means of nut "a". A PTFE gasket is necessary. The vacuum line is attached to the pumping and G-74 manifold line. A PTFE gasket is also needed here.	3. _____
4. The system is evacuated, and purged a few times with G-74.	4. Complete evacuation is necessary, and purging will tend to remove any moisture which may be in the system.
5. The system is tested, first for leaks, by introducing 15 pounds pressure, of G-74 and shutting valve "c". No drop in pressure as indicated by gauge "e" shows the system to be leak tight.	5. As pointed out in the body of this report, a leak tight system is necessary.
6. Next, the valves are tested. Fill the complete system with 15 pounds of G-74, close the Harshaw valve and valve "e". Evacuate the system, then close valve "c". Open valve "e", then the Harshaw valve. A good valve is indicated by the pressure changes as shown by the gauge.	6. In any liquid transfer system the valves are more prone to freezeing than in a vapor transfer system. This test will indicate the condition of the valves.
7. A scale of at least 25 pounds capacity is placed under the Harshaw cell, and the tare weight noted.	7. The scales will enable one to control the amount of sample taken.
8. The complete system is evacuated, and valves "e" and "c" closed. The drum valve is opended very rapidly.	8. Too slow opening of the drum valve is apt to cause the formation of a vapor block in the system. The gauge must be closed, for the pressure of the liquid C-616 exceeds the range of the gauge.

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Operation	Reason for Operation
9. At this point it is necessary to watch very carefully the weighing scales. Start closing the drum valve when the scales indicate that the Harshaw cell has about 1/2 pound less than the desired amount. (Note: If the samples to be used for freezing point determination, fluorocarbon determination, isotopic assay, and chemical assay, then 2.5 to 4.5 pounds must be taken If only the last three determinations are to be made, only a 1 pound sample is needed.)	9. It has been found that as much as 5 pounds of C-616 will flow into the Harshaw cell in 10 seconds. Great care must therefore be used in filling the cell.
10. When the drum valve "e" is closed, allow the system to drain into the Harshaw cell.	10. It is desirable to have as little C-616 lost to the pumps as possible.
11. Close the Harshaw Valve. Slowly crack the vacuum valve "c", and thoroughly pump out the system. It has been found that about seven purges with G-74 are necessary to clean out the system. While purging, have valve "e" open.	11. The large surface area of the sylphon hose necessitates a number of purges to thoroughly cleanse the system.
12. Bring the system to atmospheric pressure, and remove the Harshaw cell at the Nickel union of the adapter. If C-616 is evident, rapidly remove the Harshaw cell and blow out the system with G-74. The ventilation in the K-131 building is very adequate to safely do this.	12. Frequently small lumps of C-616 will stay in the adapter or the sylphon, and these must be removed. However, thorough purging should take care of this excess material.
13. The system is disconnected from the C&CCC drum, and the drum's valve cap and shield replaced. The drum is hoisted from the rack, and chalk-marked "Sampled" with the date, and initials of sampler.	13. _____

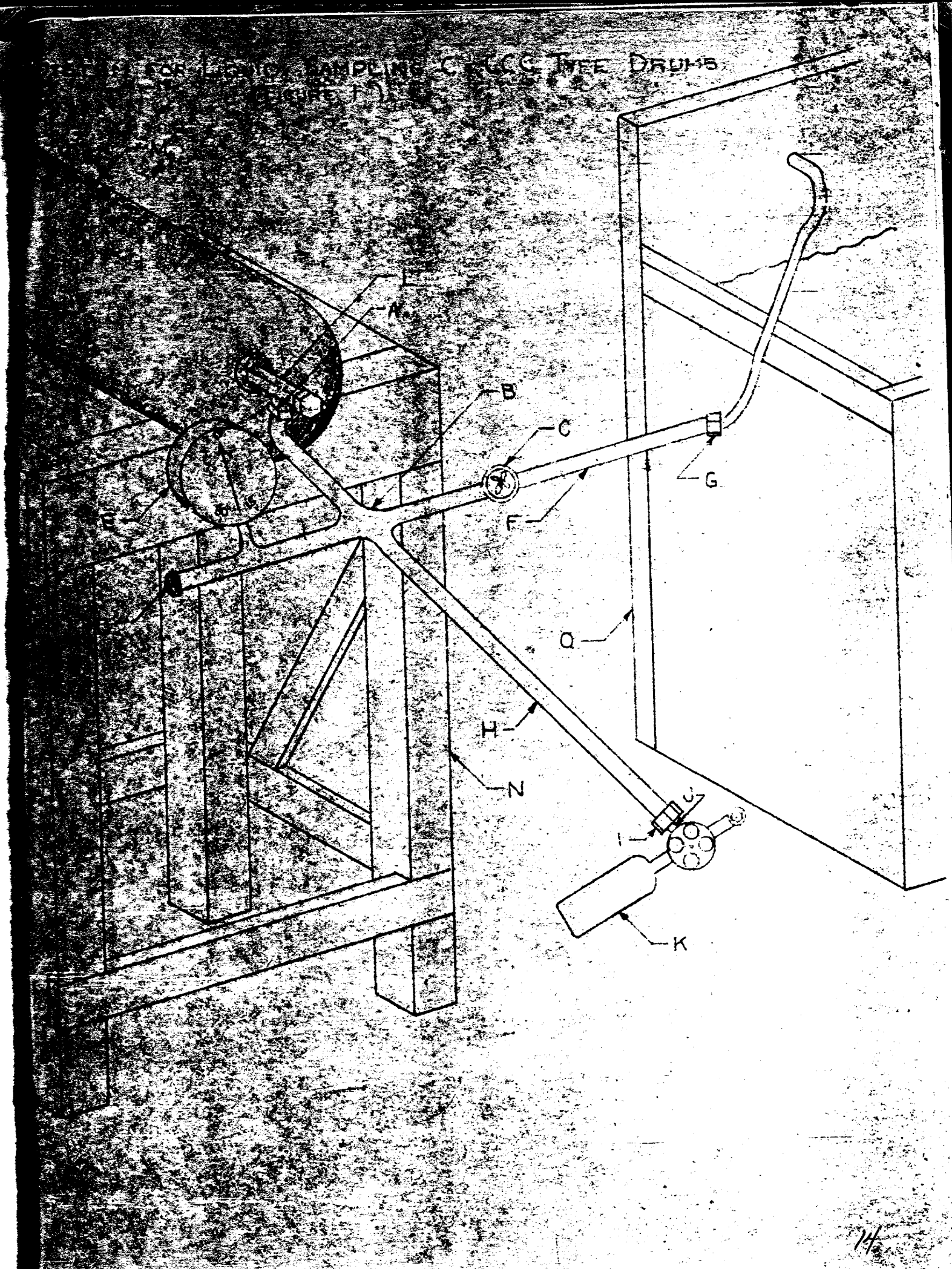
B. Suggested safety equipment to be used when liquid sampling.

1. All operators should be supplied with and should wear safety glasses.

2. All operators handling C&CCC type drums should have safety shoes in the event that a drum falls.

3. All operators should have tough, durable gloves for handling the hot cells and system.

4. At least one Chemical Warfare type gas mask should be within easy reach of the operators in case a break occurs which must be handled immediately.


L
K
B
C
G
F
E
A
H
N
I
J
K

...STEM FOR LIQUID SAMPLING "A" TYPE DRUMS
(FIGURE 2-1